

# Demonstration of NFS DeHg Process for Stabilizing Mercury (<260 ppm) Contaminated Mixed Waste

Mixed Waste Focus Area



*Prepared for*  
**U.S. Department of Energy**  
Office of Environmental Management  
Office of Science and Technology

September 1999

# Demonstration of NFS DeHg Process for Stabilizing Mercury (<260 ppm) Contaminated Mixed Waste

OST Reference #2229

Mixed Waste Focus Area



*Demonstrated at*  
Nuclear Fuel Services, Inc.  
Erwin, Tennessee



## ***Purpose of this document***

Innovative Technology Summary Reports are designed to provide potential users with the information they need to quickly determine if a technology would apply to a particular environmental management problem. They are also designed for readers who may recommend that a technology be considered by prospective users.

Each report describes a technology, system, or process that has been developed and tested with funding from DOE's Office of Science and Technology (OST). A report presents the full range of problems that a technology, system, or process will address and its advantages to the DOE cleanup in terms of system performance, cost, and cleanup effectiveness. Most reports include comparisons to baseline technologies as well as other competing technologies. Information about commercial availability and technology readiness for implementation is also included. Innovative Technology Summary Reports are intended to provide summary information. References for more detailed information are provided in an appendix.

Efforts have been made to provide key data describing the performance, cost, and regulatory acceptance of the technology. If this information was not available at the time of publication, the omission is noted.

All published Innovative Technology Summary Reports are available on the OST Web site at <http://OST.em.doe.gov> under "Publications."

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## SECTION 1

# SUMMARY

### Technology Summary

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Mercury-contaminated wastes in many forms are present at various U. S. Department of Energy (DOE) sites. Based on efforts led by the Mixed Waste Focus Area (MWFA) and its Mercury Working Group (HgWG), the inventory of wastes contaminated with < 260 ppm mercury and with radionuclides stored at various DOE sites is estimated to be approximately 6,000 m<sup>3</sup> (Conley, Morris, Osborne-Lee, and Hulet 1998). At least 26 different DOE sites have this type of mixed low-level waste in their storage facilities. Extraction methods are required to remove mercury from waste containing >260 ppm levels, but below 260 ppm Hg contamination levels, the U. S. Environmental Protection Agency (EPA) does not require removal of mercury from the waste. Steps must still be taken, however, to ensure that the final waste form does not leach mercury in excess of the limit for mercury prescribed in the Resource Conservation and Recovery Act (RCRA) when subjected to the Toxicity Characteristic Leaching Procedure (TCLP). At this time, the limit is 0.20mg/L. However, in the year 2000, the more stringent Universal Treatment Standard (UTS) of 0.025 mg/L will be used as the target endpoint.

Mercury contamination in the wastes at DOE sites presents a challenge because it exists in various forms, such as soil, sludges, and debris. Stabilization is of interest for radioactively contaminated mercury waste (<260 ppm Hg) because of its success with particular wastes, such as soils, and its promise of applicability to a broad range of wastes. However, stabilization methods must be proven to be adequate to meet treatment standards and to be feasible in terms of economics, operability, and safety. To date, no standard method of stabilization has been developed and proven for such varying waste types as those within the DOE complex.

The MWFA is investigating possible stabilization methods for mercury-contaminated mixed waste streams and has funded demonstrations, several of which have been completed. The Technology Development Requirements Document (TDRD), developed by the MWFA, requires that the effectiveness of newly developed technologies be proven. New technology for mercury stabilization must adequately stabilize waste to the new UTS, and must provide measuring and monitoring methods for verify the process. In addition the new process should:

- it must minimize worker exposure,
- it must minimize volume increase as waste is treated,
- it must minimize secondary waste generation,
- it must maximize operational flexibility and radionuclide containment.

This report summarizes the findings from a stabilization technology demonstration conducted by Nuclear Fuel Services, Inc., (NFS) under MWFA sponsorship.

### Demonstration Summary

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The MWFA supported three demonstrations to determine commercial capabilities in the field of mercury-contaminated waste stabilization. Two vendors, Allied Technology Group (ATG) and NFS, conducted demonstrations of their technologies in response to the MER02 Request for Proposal (RFP). GTS Duratek demonstrated their stabilization process in a treatability study funded principally by the Los Alamos National Laboratory (LANL), and partially by the MWFA to ensure that sufficient data could be gathered on the process to fully evaluate it. The NFS demonstration of mercury waste stabilization is the primary focus of this report. The ATG and Duratek demonstrations are reported elsewhere (MWFA 1999A, MWFA 1999B).



NFS demonstrated a process technology called DeHg<sup>SM</sup> (de-merk) on a specimen of legacy waste derived from the DOE gaseous diffusion plant located in Portsmouth, Ohio. The demonstration was performed at the NFS Applied Technology Development Laboratories located at the NFS site in Erwin, Tennessee. NFS has extensive experience with mercury hazardous waste, which helped them make their selection of test parameters, reagent dosages, and processing equipment.

The goal of the demonstration was to stabilize mercury and other RCRA metals in the mixed-waste specimen to meet UTS limits. The specimen was comprised of 30 kg of spent ion exchange resin, contaminated with mercury and radionuclides, including technetium-99 and uranium isotopes. Initial TCLP tests resulted in leachate mercury concentrations well above the UTS limit (0.49 mg/L). Scoping tests with 1 kg quantities of resin were conducted to identify optimum processing parameters. TCLP results for the scoping tests indicated that <0.005 mg/L could be achieved. Two demonstration runs with 14-kg quantities of resin were then performed. The first demonstration run achieved all UTS criteria with the exception of chromium (1.2 mg/L). The failure to meet the test criterion for chromium was attributed to the presence of chromium in the native resin water, which was used to perform the tests. The second demonstration run used a modified method for stabilizing the chromium and all UTS criteria were achieved.

## Key Results

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The key results of the demonstration are as follows:

- NFS succeeded in demonstrating a process for stabilizing the mercury-contaminated mixed waste specimen to meet all UTS TCLP limits.
- The waste form produced was a damp paste, with no free-standing water.
- No significant volume increase was observed.
- Secondary waste generation was not reported by NFS.
- Chromium with the resin slurry was more difficult to stabilize than expected using standard reduction/precipitation techniques and a modified technique was necessary for success.

## Contacts

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This ITSR was prepared by the MWFA Mercury Working Group and Prairie A&M University.

All published ITSRs are available at <http://em-50.em.doe.gov>.



## SECTION 2

### TECHNOLOGY DESCRIPTION

#### Overview

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The MWFA's HgWG has identified approximately 8,000 m<sup>3</sup> of mercury-contaminated mixed low-level and transuranic wastes in the DOE complex. In addition to elemental mercury, these waste streams include sludges, soils, and debris waste with mercury concentrations ranging from less than 2 ppm to greater than 50,000 ppm. Approximately 6,000 m<sup>3</sup> of these wastes are contaminated with <260 ppm mercury. RCRA regulations require that mercury in wastes with contamination levels at or above 260 ppm Hg be recovered by a thermal process, such as retorting, and stabilized using an amalgamation process. No treatment method is specified for wastes containing <260 ppm. However, RCRA regulations require that such wastes that exceed a mercury concentration<sup>1</sup> of 0.20 mg/L be treated by a suitable method to meet this standard.

The HgWG conducted a source selection for vendors to participate in demonstrations of different types of technologies capable of stabilizing wastes containing <260 ppm of mercury to meet the TCLP limit. Until recently, no studies beyond bench scale had been conducted on the amalgamation and stabilization of mercury mixed wastes. The primary technical issue associated with the treatment of such waste was related to scale-up of the process to a cost-effective operations level. However, the HgWG now reports the completion of three technology demonstrations on the stabilization of mixed wastes contaminated with mercury at levels <260 ppm.

ATG has recently applied its stabilization process, employing bench- and demonstration-scale processes to treat an ion exchange resin from the Portsmouth Gaseous Diffusion Facility (PORTS). NFS, located in Erwin, Tennessee, also demonstrated its stabilization process on PORTS ion exchange resin. Duratek, located in Oak Ridge, Tennessee, treated several drums of sludge and laboratory residues from LANL. The ATG and Duratek technology demonstrations are each reported in separate ITSRs (MWFA 1999A, MWFA 1999B). The NFS technology demonstration is the focus of this report. Other recent ITSRs address related issues, including: (1) treatment technologies for the amalgamation of radioactive elemental mercury (MWFA 1998A, MWFA 1998B) and (2) the effects of speciation on the stabilization of mercury wastes (Osborne-Lee 1999).

#### NFS Process Definition

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DeHg is an ambient-temperature, chemical process that converts the mercury component in mixed waste to a nonhazardous final waste form suitable for land disposal. The process was developed by NFS to address elemental, ionic, and complexed forms of mercury in mixed waste. NFS has applied the chemistry specific to their process over a variety of processing configurations for different waste matrices (i.e., shred/slurry treatment for debris, damp blending treatment for soils, decontamination treatment for nonshreddable debris, and batch treatment of bulk elemental mercury).

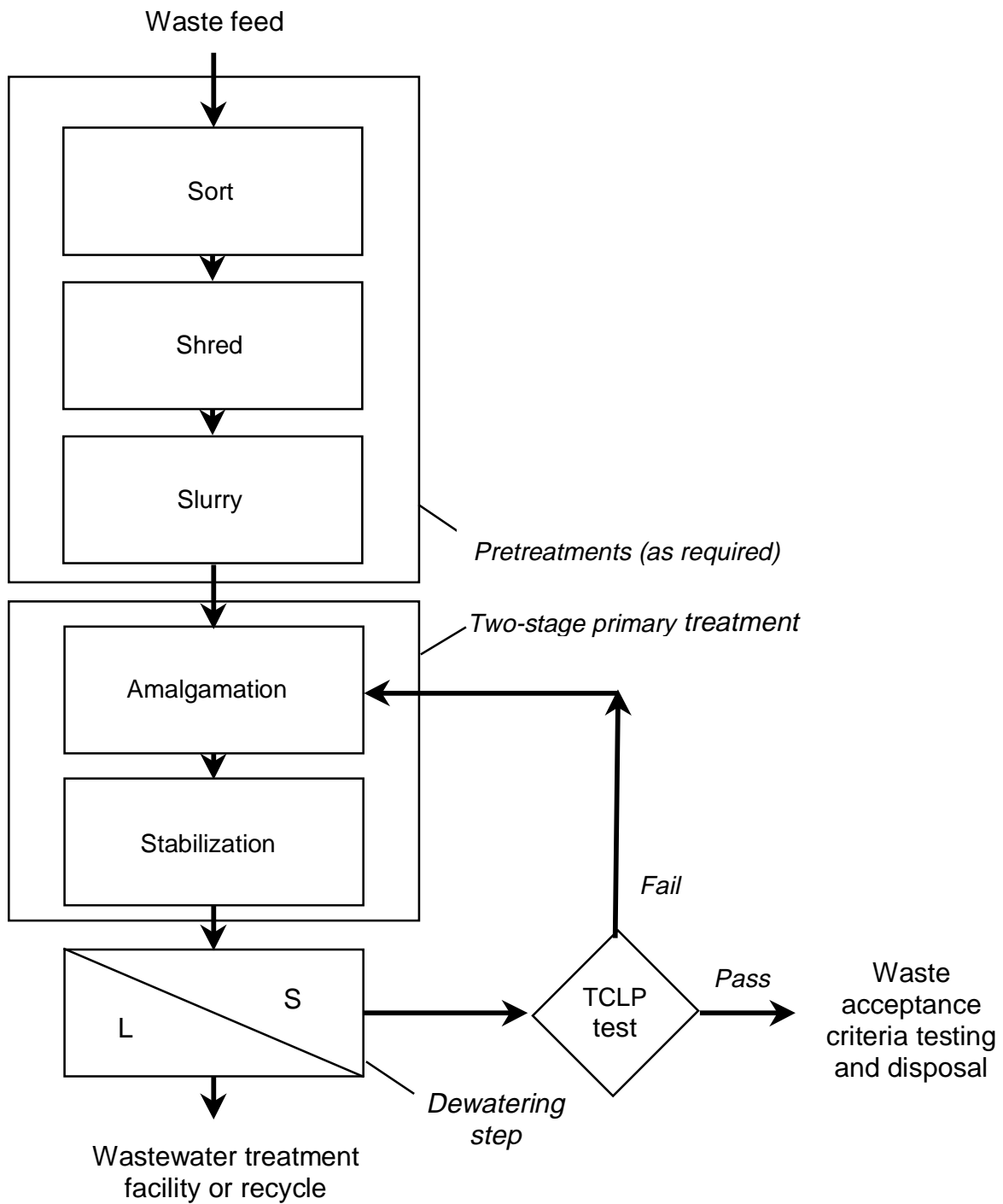
The goal of this treatability study was to demonstrate the effective stabilization of mercury and other metals on an ion-exchange resin. Effectiveness was judged by the ability of the process to produce treated sample residues, with TCLP leachate mercury concentrations less than 0.025 mg/L and other RCRA metals below UTS levels.

The NFS process consists of a two-stage treatment train that addresses the treatment of elemental or ionic mercury species alone or in combination. The general features of the DeHg process are depicted in Figure 1. The process uses standard equipment connected in typical fashion. The first stage of the process involves amalgamation of the elemental mercury component (if present). Before amalgamation, sample preparation (shredding, grinding, or slurring) may be necessary, depending on the capability of the mixing equipment to be used. The second stage of the process is the stabilization of soluble mercury species using a proprietary reagent. This reagent has the capability to free mercury from stable, soluble

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<sup>1</sup>As determined by Environmental Protection Agency (EPA) SW-846 Method 1311 Toxic Characteristic Leaching Procedure (TCLP).





**Figure 1. Block flow diagram of NFS DeHg process.**



complexes and subsequently allow for its precipitation as a stable, nonleachable salt. The DeHg reagent was originally developed to treat solidified mercuric thiocyanate wastes when traditional mercury stabilization techniques failed to treat this difficult complex.

Following treatment using the proprietary reagent(s), the residues are dewatered as needed and packaged for burial. The filtrate from this dewatering process typically contains mercury at levels less than 0.025 mg/L. This stream is reusable in the process unless overriding factors, such as criticality safety, dictate discharge of the process water. For the samples tested in this demonstration, the reagents were applied and the samples mixed as slurry as described above. Filtration of this slurry produced a damp final residue having no freestanding water. Because no free elemental mercury was present in this sample, the amalgamation portion of the process was not performed.

## **NFS System Operation**

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NFS conducted a treatability study using a bench-scale, stirred-tank reactor, closely simulating processing conditions expected for the NFS Mercury Mixed Waste Treatment Facility described above. The process is shown in Figure 1 and consists of simple procedures. No operational problems were encountered during the course of the bench-scale demonstration.

In full-scale operation, the greatest safety concerns with processing the ion-exchange resin are (1) the radioactive component of the waste and (2) potential exposure of the operating staff to mercury. To address these concerns, the NFS facility uses a fully ventilated system designed to mitigate potential emission of radioactive particulate matter. The ventilation system also has mercury-vapor-removal capability. Because the majority of the process equipment is either contained or under ventilation, and the process is operated under ambient conditions, mercury emissions are minimal.

The reagents used for this process are prepared from commercially available sources and can be safely handled using standard industrial safety practices. Chemical addition was made by manual transfer through an opening in the top of the tank.



## SECTION 3

# PERFORMANCE

### Demonstration Plan

The purpose of this study was to demonstrate stabilization of an ion-exchange resin contaminated with radioactive technetium and mercury using the DeHG process. Samples were prepared and tested according to the NFS document, *Demonstration Plan: Stabilization Process For Radioactively Contaminated Mercury Wastes, <260 PPM (MER02)*. The September 1997 demonstration plan was developed according to the specifications of the LMERC Statement of Work (SOW) and subsequent guidance. The overall protocol for testing is summarized in Figure 2.

NFS sought to stabilize mercury and other metals contained in the resin sample. Treatment effectiveness was determined based on evaluations using the TCLP protocol and its ability to achieve Waste Acceptance Criteria (WAC). The treatment goal was to produce residues having TCLP leachate mercury concentrations less than 0.025 mg/L, and to achieve UTS limits for all other metals listed in the PORTS characterization data. The UTS for mercury, now scheduled to be adopted in the year 2000, is nearly an order of magnitude lower than the present limit of 0.2 mg/L for mercury in a TCLP leachate. The EPA recently promulgated the Phase IV final ruling for Toxicity Characteristic (TC) concentration standards for toxic metals and underlying hazardous constituents (UHCs). This new ruling has established a new set of UTS limits for the standard RCRA metals and UHCs. A list of the TC, UTS, and Phase IV UTS limits are provided in Table 1. The UTS for mercury did not change, so the goal of 0.025 mg/L used in this demonstration is consistent with the new regulatory standards.

**Table 1. Summary of TC<sup>a</sup> and UTS<sup>b</sup> limits for mercury and co-contaminants**

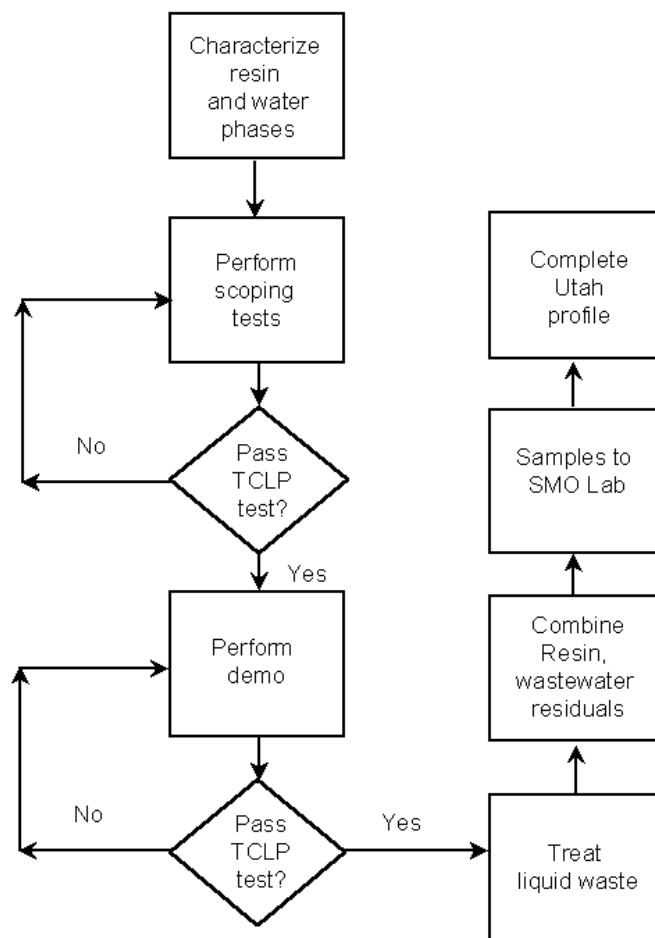
Metal	TC mg/L	Existing UTS mg/L	Phase IV UTS mg/L
Arsenic	5.0	5.0	5.0
Barium	100	7.6	21.0
Cadmium	1.0	0.19	0.11
Chromium	5.0	0.86	0.60
Lead	5.0	0.37	0.75
Mercury	0.2	0.025	0.025
Selenium	1.0	0.16	5.7
Silver	5.0	0.30	0.14
Antimony	<sup>c</sup>	2.1	1.15
Beryllium	<sup>c</sup>	0.014	1.22
Nickel	<sup>c</sup>	5.0	11.0
Thallium	<sup>c</sup>	0.078	0.20
Vanadium	<sup>c</sup>	0.23	1.6
Zinc	<sup>c</sup>	5.3	4.3

<sup>a</sup>Toxicity characteristic (TC).

<sup>b</sup>Universal treatment standard (UTS).

<sup>c</sup>TC limit not established for these underlying hazardous constituents (UHCs).





**Figure 2. General demonstration protocol for NFS DeHg process.**

Table 2 shows a characterization summary of the ion-exchange resin material, based on a revised characterization provided to NFS by the HgWG in an amendment to the SOW. Technetium-99 is the primary radionuclide of concern and mercury is the hazardous constituent of primary concern. The other constituents shown in Table 2 were assumed to be the only UHCs present in the sample for the NFS demonstration.

**Table 2. Analysis of PO-W018 ion-exchange resin used by NFS in MER02 demonstration<sup>a</sup>**

Constituent	Concentration	Units
Mercury	0.02 to 5.0	mg/L TCLP
Silver	0.02 to 0.5	mg/L TCLP
Arsenic	0.5 to 2.0	mg/L TCLP
Selenium	0.1 to 1.0	mg/L TCLP
Barium	0.03 to 14.0	mg/L TCLP
Cadmium	0.02 to 0.77	mg/L TCLP
Chromium	0.02 to 2.28	mg/L TCLP
Lead	0.2 to 3.0	mg/L TCLP
Uranium	0.4 to 3,600	ppm (total)
Technetium-99	2,300 to 14,000,000	pCi/g

<sup>a</sup>Analysis provided to NFS by PORTS via Lockheed Martin Energy Research Corporation (HgWG).

Before testing, waste samples were prepared for both onsite TCLP evaluations by NFS and offsite TCLP evaluations by Core Laboratories in Casper, Wyoming. Samples from each test batch were also evaluated in the same manner. Table 3 provides the sampling and analysis matrix for this project.

**Table 3. Sampling and analysis matrix for Nuclear Fuel Services demonstration**

Description	Number of samples	Sampling technique	Offsite analyses <sup>a</sup>	NFS <sup>b</sup> Analyses
Initial characterization of resin	2	composite and split	Full TCLP <sup>c</sup> ; Tc-99; U isotopic	TCLP metals
Initial characterization of native solution	2	aliquot	Total metals; Tc-99; U isotopic	Total Hg
Stabilized solids (process control)	2 each batch	grab	None	TCLP metals
Process Filtrates	1 each batch	aliquot	Tc-99	Total Hg
Stabilized solids (preacceptance)	2	composite and split	Full WAC <sup>d</sup> profile; Tc-99; U isotopic	none

<sup>a</sup>Performed by Core Laboratories, a Sample Management Office approved laboratory located in Casper, Wyoming.

<sup>b</sup>Nuclear Fuel Services, Inc. (NFS), located in Erwin, Tennessee.

<sup>c</sup>Toxicity characteristic leach procedure (TCLP).

<sup>d</sup>Waste Acceptance Criteria (WAC).



## Demonstration Results

### Sample Preparation and Initial Characterization

Before treatability testing, the ion-exchange resin was separated from its native water by filtering. The separated streams consisted of 12 L of solution and 30 kg of resin. The resin was evaluated per TCLP protocol. The TCLP leachate mercury concentration of the resin was 0.49 mg/L. The separated native solution was analyzed for total mercury content. This solution contained 10.4 mg/L mercury and had a pH value of 10.

### Sample Analysis Results

The offsite laboratory produced the following TCLP results (Table 4) on duplicate samples of resin:

**Table 4. Certified laboratory characterization of ion-exchange resin used by Nuclear Fuel Services<sup>a</sup> in MER02<sup>b</sup> demonstration**

Characteristic	Sample #1 TCLP <sup>c</sup>	Sample #2 TCLP	UTS <sup>d</sup> Limit <sup>e</sup>	Native Water #1	Native Water #2
Arsenic, mg/L	<0.08	<0.08	5.0	<0.08	<0.08
Barium, mg/L	1.61	1.62	21.0	0.15	0.12
Cadmium, mg/L	0.26	0.23	0.11	<0.02	<0.02
Chromium, mg/L	0.13	0.12	0.60	2.43	2.08
Lead, mg/L	<0.08	<0.08	0.75	<0.08	<0.19
Mercury, mg/L	0.36	0.36	0.025	9.6	9.2
Selenium, mg/L	<0.2	<0.2	5.7	<0.2	<0.2
Silver, mg/L	<0.05	<0.05	0.14	0.09	<0.05
Nickel, mg/L	4.35	4.08	11.0	<0.05	<0.05
Zinc, mg/L	1.30	1.21	4.3	0.10	0.07

<sup>a</sup>Nuclear Fuel Services, Inc. (NFS), located in Erwin, Tennessee.

<sup>b</sup>MER02 is a set of mixed waste treatment technology demonstrations for <260 ppm Hg contamination.

<sup>c</sup>Toxicity characteristic leach procedure (TCLP).

<sup>d</sup>Universal Treatment Standard (UTS), limits promulgated by the U.S. Environmental Protection Agency.

<sup>e</sup>Nonwastewater.

The offsite laboratory assays indicated that the levels for both cadmium (D004) and mercury (D009) exceeded the revised UTS limits for solid (nonwastewater) waste.

### Scoping Tests

A scoping test was performed using 1,000 g of resin to evaluate the proposed processing parameters for this material. Reagent grade water was used to produce the resin slurry for the treatability scoping test. The TCLP leachate mercury concentration for the treated sample was <0.005 mg/L, which was well below the UTS TCLP limit of 0.025 mg/L for mercury. All other RCRA metals were measured at less than the respective UTS levels.

The resulting resin slurry was difficult to filter due to the presence of orange fines from the resin. Addition of commercially available filtration aids to the slurry, as well as pretreatment of the filter media with the filtration aid, improved filtration rates considerably.

### Demonstration

Based on the results of the scoping tests, NFS split the remaining resin into two equal aliquots of 14 kg and subsequently applied the larger-scale DeHg process for the demonstration. NFS used native water to produce the resin slurry for the demonstration testing, thereby inputting all of the original waste to the process. Members of the HgWG and EPA representatives from the Office of Solid Waste observed the second and final demonstration test in Erwin, Tennessee, on August 20, 1998.



The NFS TCLP leachate mercury concentrations for the two proving tests averaged 0.021 and 0.011 mg/L for treated resin slurry. However, the TCLP leachate chromium concentration for the initial demonstration test sample was 1.2 mg/L, exceeding the UTS TCLP limit of 0.86 mg/L (0.60 mg/L Phase IV). The native water used in the demonstration was postulated to contain relatively high levels of chromium (2.2 mg/L) and was transported with the resin submitted to the TCLP tests. The demonstration test sample was resubmitted to treatability using a modified chromium reduction technique. The DeHg reagents were reapplied.

The NFS TCLP chromium concentrations for both tests using the modified technique were <0.5 mg/L. The TCLP leachate mercury concentrations of the retreated resin samples were 0.021 and 0.008 mg/L. Both the chromium and mercury results were below Phase IV UTS. Filtrates from the two demonstration runs were collected and treated by precipitation to remove remaining soluble metals. The residual water was evaporated and the resulting salts and aforementioned precipitates were collected and added to the resin as "processing residue."

Following testing, the treated resin and the residues from all of the tests were blended together. Duplicate samples of final treatability material were taken and profiled for Envirocare of Utah (EOU) parameters using an SMO-approved, Utah-certified laboratory. A summary of these assays, along with the raw resin analyses, is provided in Table 5.

**Table 5. Summary of Envirocare of Utah profile assays**

Utah parameter	Sample #1 raw	Sample #2 raw	Sample #1 treated	Sample #2 treated	Limit
Arsenic, mg/L, TCLP <sup>b</sup>	<0.08	<0.08	<0.08	<0.08	5.0
Barium, mg/L, TCLP	1.61	1.62	0.14	0.14	21.0
Cadmium, mg/L, TCLP	0.26	0.23	<0.02	<0.02	0.11
Chromium, mg/L, TCLP	0.13	0.12	<0.02	<0.02	0.60
Lead, mg/L, TCLP	<0.08	<0.08	<0.08	<0.08	0.75
Mercury, mg/L, TCLP	0.27	0.27	0.025 (0.021) <sup>a</sup>	0.011 (0.011) <sup>a</sup>	0.025
Selenium, mg/L, TCLP	<0.2	<0.2	<0.2	<0.2	5.7
Silver, mg/L, TCLP	<0.05	<0.05	<0.05	<0.05	0.14
Nickel, mg/L, TCLP	4.35	4.08	0.11	<0.05	11.0
Zinc, mg/L, TCLP	1.30	1.21	0.06	<0.03	4.3
Copper, mg/L, TCLP	1.36	1.34	0.04	0.03	None
PH	<sup>c</sup>	<sup>c</sup>	9.3	9.2	>2 & <12
Paint filter	<sup>c</sup>	<sup>c</sup>	Pass	Pass	Pass
Reactive sulfide, ppm	<sup>c</sup>	<sup>c</sup>	<10	<10	500 <sup>e</sup>
Reactive cyanide, ppm	<sup>c</sup>	<sup>c</sup>	<10	<10	10
Total organic halide (TOX), ppm	<sup>c</sup>	<sup>c</sup>	<sup>d</sup>	<sup>d</sup>	<200
Nuclides [pCi/g]					
U-234	252	481	612	362	37,000
U-235	8.3	16	13	12	770
U-238	17.2	36	37	25	330,000
Tc-99	14,800	4,860	9,670	8,420	190,000

<sup>a</sup>Nuclear Fuel Services, Inc., assays are shown in parenthesis.

<sup>b</sup>Toxicity characteristic leach procedure (TCLP).

<sup>c</sup>Analysis not required for untreated waste.

<sup>d</sup>Volatile (per SW-846-8260A) and semivolatile organic compounds (per SW-846-8270) were substituted in place of TOX. Data indicated that all constituents analyzed were not detected, with the exception of methylene chloride at 0.18 ppm.

<sup>e</sup>Target limit, not regulatory.



According to the offsite laboratory, both cadmium and mercury were above UTS limits in the untreated resin. The NFS process stabilized both mercury and cadmium to their respective UTS levels, and dramatically reduced the leachability of copper, zinc, nickel, chromium (D007), and barium (D005) within the resin. This comparison is exhibited in Figure 3. These data provide evidence of the capability of the DeHg process to address mercury and a variety of RCRA metals within mixed waste. TCLP mercury assays performed by NFS agreed well (0.021 and 0.011 mg/L) with those produced by the offsite laboratory (0.025 and 0.011). Further details of the Utah-certified assays have been reported elsewhere by NFS (NFS 1998).



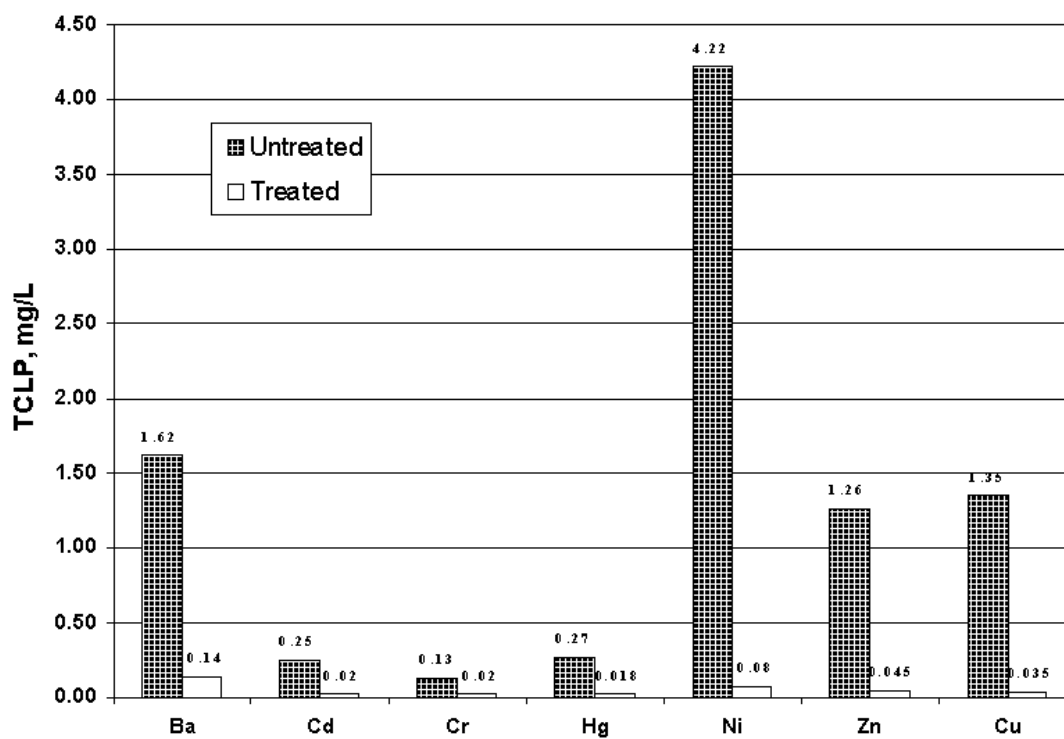


Fig. 3. NFS demonstration results: reduction of leachable metal content.



## SECTION 4

# TECHNOLOGY APPLICABILITY AND ALTERNATIVE TECHNOLOGIES

### Competing Technologies

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#### Baseline Technologies

While EPA's RMERC and IMERC regulations address mercury within waste at concentrations exceeding 260 ppm, there is relatively little work on the effective stabilization of mercury in problematic mercury contaminated mixed wastes at <260 ppm. This need has driven the mission of the MWFA to identify and validate useful industrial mercury-stabilization technologies for DOE mixed wastes. Although RMERC could be applied to mercury in the <260 ppm range, there are both public and regulatory concerns about mercury emissions. In addition, RMERC alone does not address the potential need to stabilize thermal residuals before disposal in a licensed and permitted landfill. Hence, there is no real baseline technology for mercury-contaminated mixed wastes at <260 ppm, although several promising new and developing technologies do exist.

Sulfur polymer cement offers some potential for mercury stabilization. However, this process is sensitive to water content of the subject material and requires elevated temperature for application. For the specific matrix tested in this work, sulfur polymer cement would be as useful as other competing technologies, such as the NFS DeHg process, given the high water content and the relatively low decomposition temperature of ion-exchange material.

The MER02 SOW specified technology demonstrations to address the deficiencies in technology for treating mercury-contaminated mixed waste. Under the MER02 SOW, two vendors were funded to demonstrate their newly-developed/developing processes on actual mixed wastes. In addition, Los Alamos issued a contract to GTS Duratek to perform a treatability study on other waste. The MWFA added to that contract to have Duratek provide similar information to that being collected in the MER02 demonstrations. The processes demonstrated by ATG, Duratek, and NFS represent competing technologies. As newly demonstrated technologies, comparisons of these processes, among themselves and with respect to the baseline, are of particular interest. Important criteria for comparison include: (1) performance in treating mercury-contaminated mixed waste (<260 ppm), (2) applicability to the target and other waste categories, (3) cost, and (4) risk to workers and the public.

Currently, however, some demonstration findings are still being gathered at the time of this report, such that a full comparison of competing technologies must await a future report. Nevertheless, important criteria for comparison are reported here for the NFS process along with similar information for ATG and Duratek, where available.

#### Technology Applicability

NFS developed its DeHg process to address a large variety of chemical forms of mercury. The process has been successfully applied to and is useful for a wide variety of waste matrices, which include:

- shreddable debris,
- nonshreddable debris,
- waste waters,
- soils,
- sludges,
- organics.



NFS used the DeHg process to successfully demonstrate treatment of elemental mercury waste in a MER01 demonstration (MWFA 1998B). NFS also applied the process to demonstrate the treatment of mixed waste containing mercury in the form of various chemical species (Osborne-Lee 1999). In a companion project to MER02 sponsored by the MWFA, NFS used the DeHg process to stabilize the following forms of inorganic, organic, and complexed mercury at <260 ppm: elemental mercury, mercuric chloride, mercuric iodide, phenyl-mercuric chloride, mercuric oxide, mercuric cyanide, and mercuric thiocyanate.

Mixed wastes with free elemental mercury will require the addition of amalgamation, as well as stabilizing reagents to address the various chemical species of mercury. Soils and sludges containing free elemental mercury represent a particular challenge. This is ascribed to potential matrix-mercury interaction effects, which render stabilization less effective. In addition, the elemental mercury contamination within these matrices is unevenly distributed. Therefore, mixing methods become important in dispersing amalgamating/stabilizing reagents to all sites within the matrix that contain the mercury contamination.

This technology, while being directed by NFS to the DOE market, also has potential commercial applications. Certain select matrices, such as sludges, soils, adsorbents, generated by commercial entities, are similar to those within DOE, except without the radiological components. One such example is chlor-alkali wastewater sludge, which may contain several forms of mercury. As such, they are considered hazardous only, not mixed. Another interesting application would be the stabilization of mercury-contaminated wastes generated during gold-mining operations.

### **Patents/Commercialization/Sponsor**

The DeHg process is proprietary to NFS. Regarding ability to permit and operate the process, NFS permitted and operated DeHg for treatment of NFS mixed waste in the State of Tennessee during 1995. NFS has since received a revised license and permit for this system in order to process other mercury mixed wastes. NFS is presently investigating the potential of siting a mercury-mixed-waste-treatment system at a Treatment, Storage, and Disposal Facility (TSDF) that is licensed and permitted for mixed waste.

## **Other Competing Technologies**

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Table 6 summarizes technology features and demonstration results for the three vendors participating in the MER02 mixed waste treatment demonstrations.

### **ATG Stabilization Technology**

ATG has demonstrated a full-scale stabilization facility that uses a dithiocarbamate (DTC) formulation to produce a stabilized waste, which satisfies the UTS treatment limits for mercury. The DTC formulations used by ATG reproducibly stabilized over 99% of the mercury initially present at levels of about 40 times the UTS in the ion exchange process stream from PORTS. Volume increases were reported to be small, at 16% of the untreated waste volume. The process in stabilizing additional constituents, namely barium, cadmium, and chromium, demonstrated some robustness for broader application.

### **GTS Duratek Stabilization Technology**

Duratek has evaluated the efficacy of Portland cement-based grout for stabilization of sludge and laboratory residues generated at LANL. Duratek performed bench-scale solidification tests at low and high waste loading. Full-scale drum-solidification capabilities were demonstrated. The Duratek demonstration was subject to complications due to unexpected characteristics of the waste stream.

The presence of organic compounds, including pesticides, in the sludge created additional processing requirements, including pretreatment such as shredding and thermal treatment. The difficulty of stabilizing wastes containing a significant amount of organic compounds is well known. The presence of unexpectedly high amounts of radioactivity in the specimen lead in the direction of high volume increases at the low-waste loading necessary to obtain land disposal of the final waste form. These uncertainties make life-cycle cost difficult to project. Likewise, the additional challenge posed by the presence of organic hazardous constituents and high radionuclide concentrations makes comparison of the Duratek technology with those of ATG and NFS unfeasible.



**Table 6. Summary of findings from tests by GTS Duratek, Nuclear Fuel Services, and ATG on the stabilization of mixed wastes (<260 ppm Hg)**

Comparison factor	GTS Duratek, Inc.	Nuclear Fuel Services, Inc. (NFS)	Allied Technology Group, Inc. (ATG)
Waste type tested	Sludge and laboratory residues from Los Alamos National Laboratory	Ion-exchange resin from Portsmouth, Ohio facility	Ion-exchange resin from Portsmouth, Ohio facility
Process mechanism	In-drum mixer based operation	Standard laboratory glassware operation based in a ventilation hood	Pug mill, mortar mixer, hazardous material enclosure, with ventilated hood and air treatment system
Scale of bench test	0.25–0.35 kg bench scale tests performed	1 kg scoping tests performed	0.6 kg bench tests performed
Scale of demonstration	55-gallon drum (about 200–400 kg)	14 kg batches	33 kg batches (full-scale)
Final waste form	Fails TCLP test for organics and pesticides; fails WAC for radionuclides	Passes all TCLP and UTS tests (Hg, Cd, Cu, Zn, Ni, and Cr) but modified formulation required for Cr	Most effective for mercury and Cr, moderately effective for Ba and Cd
Stabilization process	Portland cement-based grout	Uses proprietary formulation of additives and EPA-prescribed agents	Uses dithiocarbamate, or other nonproprietary agents, and a small amount of proprietary liquid
Effect of contaminants on the process	High concentration of organic compounds or radionuclides make effective waste form difficult to achieve	Fines from resin created filtration challenge, solved by use of filter aids	Water <10% tolerated. Other contamination not addressed
Throughput	<sup>a</sup>	1,000 lb/h, per SOW	1,200 lb/h at full scale
Cost	<sup>a</sup>	~\$6/kg at 1,000 lb/h to \$37/kg at 100 lb/h	Not yet available
Waste Acceptance Criteria	<sup>a</sup>	Both processes produce waste forms that meet current Envirocare Waste Acceptance Criteria (WAC)	
Moisture	Moisture (water in small amounts) is tolerated by all three processes		
Physical characteristics	<sup>a</sup>	Waste form characteristics are physically similar	
Regulatory and safety requirements		No additional hazards, safety, or regulatory issues found for either process	
Summary assessment	<sup>a</sup>	Less costly, better leach performance (in meeting UTS) for more UHCs, but formulations are proprietary	Higher waste loadings, less secondary waste, and fewer proprietary reagents

<sup>a</sup>Waste specimens tested by ATG and NFS are very comparable. Duratek sample was very different, thus basis for comparison of three vendor technologies is difficult to achieve. Also, uncertainties in waste characteristics complicate Duratek demonstration, delaying the availability of key results. No comparison is made of Duratek with ATG and NFS at this time.

<sup>b</sup>Universal treatment standard (UTS). Underlying hazardous constituents (UHCs). Waste acceptance criteria (WAC).



## SECTION 5

### COST

#### Cost Methodology

Using a proprietary model, NFS estimated life-cycle unit cost values for waste processed based on a “tiered” approach. The tiered approach for presenting cost is based on four throughput rates, with 1,000 lb/h as the maximum rate for treatment of mercury mixed waste. The life-cycle design and cost basis are summarized in Table 7.

**Table 7. Life-cycle design and cost basis for processing mercury mixed waste**

Parameter	Design and Cost Basis
Plant life	10 years
Operations	250 days/year, 5 days/week, 8 h/d
Throughput cases	1,000 lb/h, 500 lb/h, 250 lb/h, 100 lb/h
Battery process	Installed at existing TSDF
Capital costs	Engineering, equipment, construction, licensing, permitting, testing, and startup
Treatment costs	Personnel, supplies, analytical, wastewater, utilities, depreciation, benefits, general and administrative, maintenance and repairs; transportation and disposal costs not included.
Decommissioning costs	Decontamination, decommissioning, waste disposition, and required environmental restoration

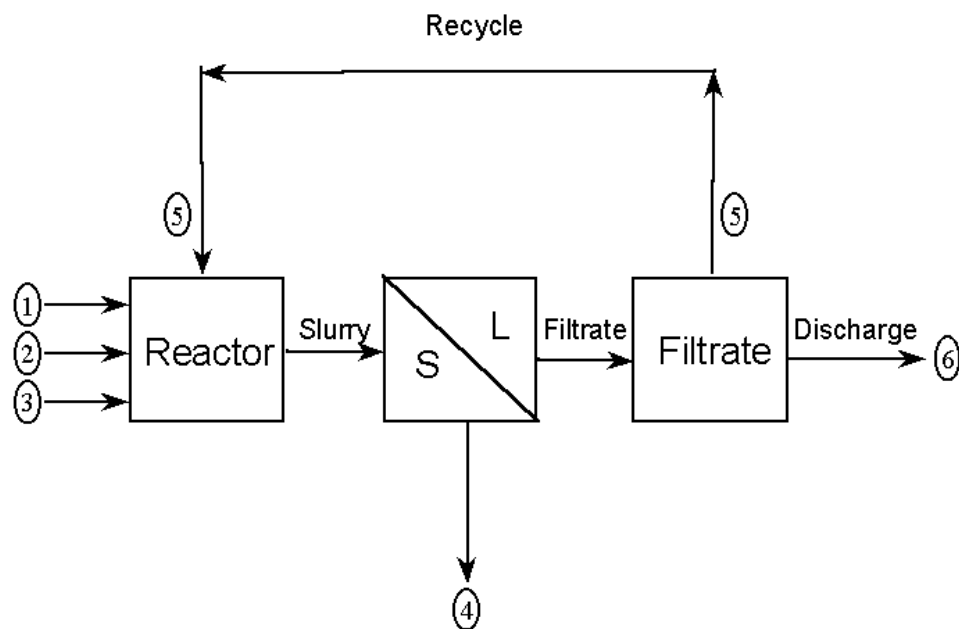
A simplified block flow diagram and material balance based on the demonstration testing is presented in Figure 4. The waste feed rate to the system is assumed to be 1,000 lb/h. A key feature of the process is that the filtrate may be recycled to the reactor and added to the waste feed to produce a slurry for treatment. Filtrate may be retreated further, if necessary, to meet discharge criteria for metals; however, NFS projects based on their experience that such treatment will not normally be required due to the efficiency of the DeHg reaction.

#### Cost Conclusions

For the steady-state 1,000 lb/h processing rate specified in the original solicitation for this work, the costs ranged from \$5.35/kg to \$6.93/kg for soils, sludges, and shreddable debris under 5 wt% in mercury concentration. These costs escalate to \$33/kg to \$37/kg in the same mercury concentration range for processing rates averaging 10% of the specified steady-state rate (100 lb/h). Calculations were also made for intermediate processing rates, as well as for wastes containing differing concentrations of mercury. Costs do not include either transportation or disposal of waste.

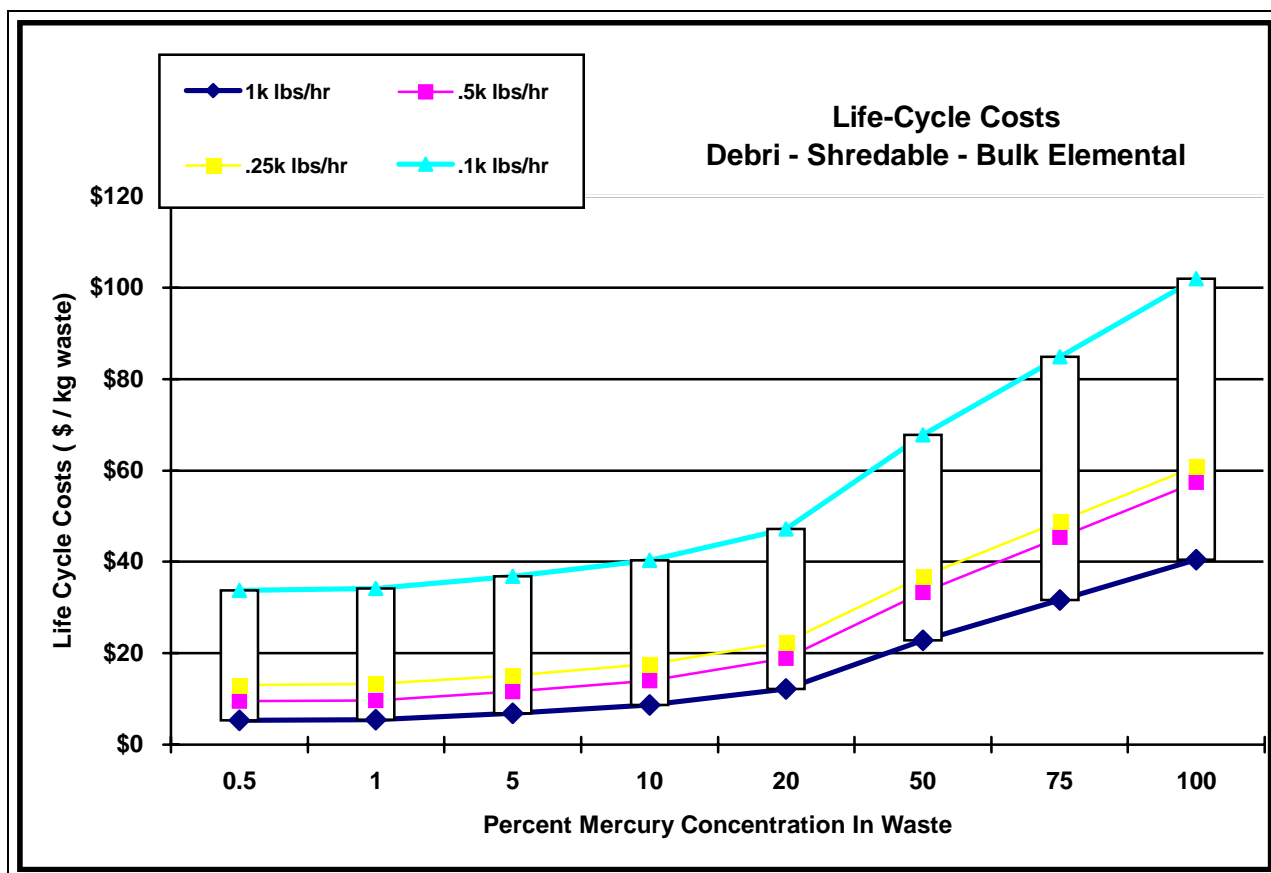
Figure 5 illustrates the results of the life-cycle unit cost model. NFS reports these costs to be conservative, while indicating that actual costs will depend upon the: (1) homogeneity of the waste, (2) nature of the matrix being processed, and (3) presence of other hazardous constituents requiring treatment.





Stream #	Name	lb.
1	Waste	1,000
2	Makeup Water	200
3	Reagents	100
4	Treated Solids	1,300
5	Recycle Water	1,000
6	Discharge Water	1,000

Figure 4. Block flow diagram and material balance for NFS DeHg process.



**Figure 5. Life-cycle unit cost of treating mercury mixed waste with NFS DeHg process.**

## SECTION 6

### REGULATORY/POLICY ISSUES

#### Regulatory Considerations

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The regulatory and permitting issues related to the use of stabilization technology for treatment of mercury contaminated wastes are governed by the following safety and health regulations:

- Occupational Safety and Health Administration (OSHA), 29 CFR 1926
  - 1926.28 Personal Protective Equipment
  - 1926.102 Eye and Face Protection
  - 1926.103 Respiratory Protection
- OSHA 29 CFR 1910
  - 1910.132 General Requirements (Personnel Protective Equipment)
  - 1910.133 Eye and Face Protection
  - 1910.134 Respiratory Protection

Disposal requirements/criteria include the following Department of Transportation (DOT) and DOE requirements:

- 49 CFR, Subchapter C, Hazardous Materials Regulation
  - 171 General Information, Regulations, and Definitions
  - 172 Hazardous Materials Table, Special Provisions, Hazardous Materials Communications, Emergency Response Information, and Training Requirements
  - 173 Shippers—General Requirements for Shipments and Packaging
  - 174 Carriage by Rail
  - 177 Carriage by Public Highway
  - 178 Specifications for Packaging
- 10 CFR 71 Packaging and Transportation of Radioactive Material

If the waste is determined to be hazardous solid waste, the following EPA requirement should be considered:

- 40 CFR, Subchapter 1 Solid Waste

#### CERCLA Criteria

The Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) has established nine criteria against which alternative treatment approaches are to be judged during the Remedial Investigation/Feasibility Study (RI/FS) portion of the remediation action. A short explanation of each of the criteria (EPA 1988) and the assessment of the NFS process against it follows.

##### Overall protection of human health and the environment

This criterion is an evaluation of the overall protectiveness of an alternative, should focus on whether a specific alternative achieves adequate protection, and should describe how site risks posed through each pathway being addressed by the FS are eliminated, reduced, or controlled.

In a CERCLA environment, the resulting waste forms from the NFS process will provide improved protection of human health and the environment by reducing the mobility of the elemental mercury. The amalgams should be placed inside another container to further enhance protection.



### Compliance with ARARs

This evaluation criterion is used to determine whether each alternative will meet all of its federal and state Applicable or Relevant and Appropriate Requirements (ARARs) that have been identified in previous stages of the RI/ FS process.

The LDRs are the most likely ARAR to be applied to a CERCLA site dealing with mercury wastes. Regulations under RCRA specify no standard treatment for elemental mercury. The NFS process provides a potential future best practice for this waste type.

### Long-term effectiveness and permanence

Alternatives under this criterion are to be evaluated in terms of risk remaining at the site after response objectives have been met. The primary focus of this evaluation is the extent and effectiveness of the controls that may be required to manage the risk posed by treatment residuals and/or untreated wastes.

The long-term effectiveness of any remediation process has to be judged not only by the efficacy of the actual treatment process, but by how well the process can be applied to the extent of the contamination. Assuming that the mercury waste can be efficiently brought to NFS's equipment, the process should be able to provide environmental protectiveness. Tests to be performed at the Oak Ridge National Laboratory (ORNL) will provide a more definitive answer.

### Reduction of toxicity, mobility, or volume through treatment

The statutory preference for selecting remedial actions that employ treatment technologies that permanently and significantly reduce toxicity, mobility, or volume of the hazardous substances are to be evaluated under this criterion.

Stabilization with the NFS process, or a similar process, should significantly reduce mercury's mobility in a waste management scenario. In a CERCLA action, further study would be required to assess how the action of bacteria affects the waste form. Secondary containment may be prudent in any case.

### Short-term effectiveness

This criterion addresses the effects of the alternative during the construction and implementation phase until remedial response objectives are met.

As designed and operated, the NFS process should be protective of the community and the workers while not imposing meaningful environmental consequences during its operation.

### Implementability

The implementability criterion focuses on the technical and administrative feasibility of implementing an alternative and the availability of various services and materials required during implementation.

The process should prove to be viable from the standpoints of both the technical (ability to construct, reliability, and monitoring) and administrative (coordination with other agencies) feasibility, as well as the availability of services and materials.

### Cost

The costing procedures found in the *Remedial Action Costing Procedures Manual* are to be the bases for comparing alternatives with regard to costs.

The cost figures to be provided in the future were not based on the rigor detailed in the referenced document above.

### State acceptance

This assessment evaluates the technical and administrative issues and concerns the state (or support agency in the case of state-lead sites) may have regarding each of the alternatives.

See "Safety, Risks, Benefits, and Community Reaction" subsection below.





### Community Acceptance

Under this criterion, an assessment is made on the issues and concerns the public may have regarding each of the alternatives.

See “Safety, Risks, Benefits, and Community Reaction” subsection.

## **Safety, Risks, Benefits, and Community Reaction**

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Regarding acceptance by regulators and the public, DeHg has been permitted in the State of Tennessee for treating NFS production waste containing radioactively contaminated elemental mercury. NFS has since acquired a broad radioactive materials license and modified the permit to accept a variety of mercury mixed wastes, including bulk radioactively contaminated mercury, from sites other than NFS. Through this permitting and operating experience, NFS demonstrated to appropriate regulatory agencies that DeHg:

- is protective of human health and the environment through both engineering and administrative controls,
- reduces the toxicity and mobility of material through treatment,
- can be implemented, with potential for deployment at other locations;
- has been accepted by the State of Tennessee,
- has been accepted by the community as indicated in public hearings during the permitting process.

NFS is considering deployment of this technology at one or more mixed waste treatment, storage, and disposal facilities. NFS is optimistic that the experience of the two prior permitting campaigns will prove beneficial in siting the system at an offsite facility.

The MWFA, considering eight criteria for the level of risk as associated with mercury stabilization, evaluated other aspects of risk, as follows:

- correctness (technical correctness),
- cost (effectiveness to use),
- permitability (ease of permitting),
- safety,
- sponsorship (commitment by sponsors),
- completeness (ready for use),
- acceptability (to stakeholders),
- timeliness (to meet schedules).

The risk values, established for the MWFA-developed technology processes have been derived from top-level requirements defined in the MWFA Systems Requirements Document. Evaluations of the technology and assignment of risk values were made by a team comprised of HgWG members in consideration of the risk category definitions and performance observations from the demonstration experience. The assessments made are summarized below.

### **Correctness**

This risk category is moderately low. The targeted volume of waste to be treated is not large compared with most other waste types. Prior successes with low-level radioactive wastes, bench-scale tests with mixed wastes, and, now, largely promising demonstration results indicate that stabilization is a reasonable option for many mercury mixed waste streams. Limitations to stabilization as a stand-alone technology for mercury waste treatment may be overcome by using it in train with supporting treatment steps.



## **Cost**

This risk category is rated as moderate. The targeted volume to be treated is not large, but the waste possesses diverse characteristics. Oxidation, complexation, and speciation of mercury across various matrices add an element of uncertainty as to the difficulty of successfully stabilizing the bulk of inventory (and future generation) without process modifications. In addition, cost estimates provided by NFS show that stream characteristics greatly influence unit cost.

## **Permitability**

This risk category is rated as very low. The treatment process is simple and based on a well-proven Best Available Demonstrated Technology for nonradioactive mercury waste. The volumes of waste involved are not large enough to pose much likelihood of regulatory problems.

## **Safety**

This risk category is rated as low. While mercury is a hazardous material of some concern and radioactive contamination has the potential to raise additional concern, mercury vapors and leaching appear to be well controlled by the process and radioactive contamination is low. The stability of the final waste form is key in immobilizing both mercury and radionuclides, thereby minimizing concerns over worker safety, public safety, and environmental protection (Connor 1990).

## **Sponsorship**

This risk category is rated as moderately low. Interest by the sites has been good, and programmatic support for technology development has demonstrated good commitment. There is a small risk that some potential users may find a local or onsite solution for treatment of their mercury wastes.

## **Completeness**

This risk category is rated as moderately low due to the simple, proven nature of stabilization, in consideration of the potential complexity of mercury chemistry and diversity of waste matrices, especially in light of recent successful demonstrations.

## **Acceptability**

This risk category is rated as very low. Stabilization is a process easily identifiable to the public because of the widespread use of cement and concrete. The waste form stability, simplicity, and familiarity to the public that characterize the technology are expected to make for easy public acceptance.

## **Timeliness**

This risk category is rated low. Based on preliminary information received to date from 10 DOE sites, the timeframe for treatment is late FY-99 and FY-00.

## **Public Participation**

The siting of a mixed waste treatment facility of any kind near communities will involve public input. Stakeholders are generally concerned about the type, toxicity, and amount of emissions to be discharged to the atmosphere and the disposal site for the final waste form.

The MWFA Tribal and Public Involvement Resource Team and HgWG initiated activities to involve and gather stakeholder issues, needs, and concerns about mercury treatment technologies. These activities included reviews, articles, and presentations. During November and December of 1997, the chair of the HgWG addressed both the Oak Ridge Local Oversight Committee and the Site Specific Advisory Board (SSAB). The purpose of the November 17–18, 1997, meetings was to identify issues, needs, and concerns of various Oak Ridge stakeholders regarding technologies that may be applicable to Oak Ridge. The areas emphasized included continuous emission monitors, characterization, input to Technology Performance Reports, and the HgWG. These meetings were interactive, where participants explored the issues and problem-solved collectively. No formal presentations were made, but information was provided



and progress on various MWFA projects was discussed. Participants included members of the local oversight committee, the Site Technology Coordination Group (STCG), and the general public.

The SSAB Environmental Technology Group meeting on December 10, 1997, involved providing stakeholder input into various technologies development projects at Oak Ridge. Those they have expressed interest in addressing are:

- Transportable Vitrification System
- Toxic Substances Control Act (TSCA) Test Bed for Continuous Emissions Monitors
- Mercury Working Group/Mercury Treatment Demonstrations
- Removal of Mercury from Liquid Wastes.

A short presentation on the status of each activity was given and the proposed future scopes were discussed.

The MWFA assembled a Technical Requirements Working Group (TRWG), a stakeholder group capable of representing varied Tribal and public perspectives. The TRWG assisted MWFA technical staff in transforming or integrating site-specific issues, needs, and concerns into the TDRDs, and providing Tribal and public perspectives to technical staff for identifying and resolving technical issues. The TRWG reviewed and provided recommendations to the MWFA on changes to the Mercury TDRDs.

Lastly, the MWFA Resource Team facilitated tribal and public involvement by issuing an article in the quarterly, July 1997, newsletter highlighting mercury treatment and disposal.



## SECTION 7

### LESSONS LEARNED

#### Implementation Considerations

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Key lessons learned during this project were as follows:

- Fines in the untreated resin matrix provided an unexpected filtration challenge. This challenge was met through the use of commercially available filtration aids to improve filtration rates.
- This waste was originally characterized by the generator before the final Phase IV ruling, which now requires that designated metals and any UHCs be stabilized to UTS TCLP criteria. The new ruling indicates that mixed waste, even though characterized, will need to comply with this ruling when it takes effect in the year 2000.
- Chromium in the resin slurry was more difficult to treat than expected. Initial scoping tests using standard chromium reduction/precipitation techniques did not yield UTS results. The stabilization of chromium was accomplished using a modification of a standard reduction technique.
- This work shows that UTS levels in TCLP leachate for RCRA metals can be attained for this waste matrix using the DeHg process.



## Appendix A

### REFERENCES

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## Appendix B

### ACRONYMS AND ABBREVIATIONS

ADA	ADA Technologies (Englewood, Colorado)
ATG	Allied Technologies Group, Inc. (Fremont, California)
ARARs	Applicable or Relevant and Appropriate Requirements
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CFR	Code of Federal Regulations
DeHg	A proprietary process by NFS for processing mercury mixed waste (pronounced de'-merk)
DOE	Department of Energy
DOT	Department of Transportation
DTC	Dithiocarbamate, a reagent in a formula for stabilization
Duratek	GTS Duratek, Inc., (Oak Ridge, Tennessee)
EOU	Envirocare of Utah
EPA	Environmental Protection Agency
HgWG	Mercury Working Group, MWFA
INEEL	Idaho National Engineering and Environmental Laboratory
ITSR	Innovative Technology Summary Report
LANL	Los Alamos National Laboratory
LDR	Land Disposal Restrictions
MER01	A solicitation to industry (November 1996) entitled, "Demonstration of the Amalgamation Process for Treatment of Radioactively Contaminated Elemental Mercury Wastes"
MER02	A solicitation to industry (September 1997??) entitled, "Demonstration of the Stabilization Process for Treatment of Radioactively Contaminated <260 ppm Mercury Wastes"
MWFA	Mixed Waste Focus Area
NFS	Nuclear Fuel Services, Incorporated (Erwin, Tennessee)
ORNL	Oak Ridge National Laboratory
OSHA	Occupational Safety and Health Administration
PORTS	Portsmouth Gaseous Diffusion Plant
ppm	parts per million
RCRA	Resource Conservation and Recovery Act
RFP	Request for Proposal
RI/ FS	Remedial Investigation/ Feasibility Study
SOW	Statement of Work
SSAB	Site Specific Advisory Board
STCG	Site Technology Coordination Group
TC	Toxicity Characteristic
TCLP	Toxicity Characteristic Leaching Procedure
TDRD	Technology Development Requirements Document
TOX	Total organic halide
TRWG	Technical Requirements Working Group
TSCA	Toxic Substance Control Act
TSDF	Treatment, Storage, and Disposal Facility
UHC	Underlying hazardous constituent
UTS	Universal Treatment Standard
WAC	Waste Acceptance Criteria

